PATENT SPECIFICATION

901,663

Inventors: DENIS CHARLES LOWE and
ANDRES SANCHEZ CABELLO

Date of filing Complete Specification: November 10, 1960

Application Date: November 12, 1959. *No.* 38475/59

Complete Specification Published: July 25, 1962

Index at Acceptance:—**Classes 2(5)**, R1C(5:6:8:16), R2C(5:6:8:16), R6C(5:6:8:16), R13C(5:6:8:16), R29C(5:6:8:16), R30C(5:6:8); **and 140**, A(2G:2K1A:5G1A:5G1B:5G4:11F:18).

International Classification:—C08g. B29d.

COMPLETE SPECIFICATION

NO DRAWINGS

Flame Resistant Materials

WE, FORMICA INTERNATIONAL LIMITED, a Company organised under the laws of Great Britain, of De La Rue House, 84-86 Regent Street, London, W.1., do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to resinous compositions having flame resistant properties and to materials treated with such compositions.

Laminated plastics comprising fibrous materials treated with synthetic resins are widely used in the construction of furniture of all kinds and as wall panelling. Although the majority of these materias are not inflammable and do not present a fire risk in themselves, it is desirable that they should have a high flame resistance particularly when they are used as constructional elements in buildings and ships.

The object of this invention is to provide resinous compositions which, when used to treat the fibrous core materials of a laminated product, give to the laminated products flame resistant properties and more particularly, but not exclusively, Class 1 flame resistance characteristics as defined in British Standard Specification No. 476.

According to this invention a process for producing a flame resistant resinous composition consists in adding to a water-containing solution of an alkali-catalysed resinous condensation product of formaldehyde and a phenol, a neutralising agent which gives rise, on reaction with free alkali, to inorganic salts having flame resistant properties, and an amino formaldehyde resinous condensation product, the neutralising agent being added in an amount sufficient to neutralise the alkali-catalysed resinous condensation product of formaldehyde and a phenol.

The resinous condensation products thus produced are phenolic resin condensation products. Phenolic resin is defined in British Standard No. 1755/1951 under No. 1229 as "a synthetic resin produced by the condensation of a phenol with an aldehyde."

In order to obtain a product with very high flame resistant properties, it is preferred to add a water soluble inorganic salt having flame resistant properties to the mixture.

Preferably the proportion based on solids of amino formaldehyde condensate to phenolic condensate is at least one to two.

Preferably the alkali used to catalyse the phenolic condensation product is sodium hydroxide.

Preferably the neutralising agent used to neutralise the phenolic condensation product is orthoboric acid.

Preferably the amino compound used in the amino formaldehyde condensate is melamine.

The water soluble inorganic salt having flame resistant properties is preferably added in a proportion exceeding 2.5% of the total solids of the resinous composition, and is preferably sodium metaborate.

Preferably the phenolic condensation product is substantially compatible with water.

Flame resistant laminated plastics may be produced by treating some or all of the core sheets of the product, e.g. paper, fabric, asbestos or other fibrous material, with the resinous flame resistant composition before assembly and consolidation of the core and separate components of the laminates.

Preferably the core sheets contain between 33% and 38% by weight of the flame resistant resinous composition.

Although the phenolic resins may be catalysed by ammonium hydroxide, it is preferred to use those catalysed by sodium hydroxide because they are generally more

compatible with water. In the treatment of fibrous materials generally, and cellulosic materials in particular, substantially complete penetration with the resinous composition is required if they are to have uniform flame resistant properties and it is for this reason that water soluble resins are best. However, the solvent can include small quantities of methylated spirits.

The existence of free alkali in alkali-catalysed resins used in the production of laminated plastics detracts from the water resistance of the final product and for this reason it is necessary for it to be neutralised. Although a wide range of neutralising agents may be used this invention provides for the use of one which will react with the free alkali and produce an inorganic salt having flame resistant properties and hence orthoboric acid is preferred because reaction with sodium hydroxide produces soluble sodium borates. Phosphoric acid may be used but has the disadvantage of causing resin precipitation and resolublisation is necessary by the addition of methylated spirits or another suitable solvent.

It has been found necessary to add sodium metaborate to the resinous composition in order to increase the flame resistant characteristics which may not be adequately provided by the borates produced by neutralisation of the sodium hydroxide. Other suitable salts, for example sodium pentaborate, borax, disodium phosphate, mono ammonium phosphate, triethanolamine phosphate and "Polybor" (Registered Trade Mark), a proprietary product of Borax Consolidated Co. may be used either alone or in suitable admixture with each other or with sodium metaborate. Flame resistant properties of materials treated with the resinous compositions according to this invention are good, but if Class 1 flame resistant characteristics, as defined in British Standard Specification No. 476, are required it is necessary to add not less than 2.5% and preferably about 5%, of the total resin solids, of the salt having flame resistant properties. We have found that about 5% of the salt is an optimum quantity.

In order to obtain the best flame resistant properties it is necessary to include in the composition amino-formaldehyde condensation product resins because there are inherently more flame resistant than phenolic resins. Melamine formaldehyde resins are eminently satisfactory and although, for laminating compositions, the best proportion of melamine resin to phenolic resin is one part of the former to two parts of the latter, satisfactory products can be produced by using, for example, one part of melamine resin to one part of phenolic resin or even two parts of melamine resin to one part of phenolic resin.

Again although phenol formaldehyde resin is eminently satisfactory, condensation products of cresols and formaldehyde can be used, provided the degree of condensation is controlled to produce products having high water compatibility.

In a particular case a conventional phenol formaldehyde resin (1 mol phenol to 1.4 mol formaldehyde), catalysed with sodium hydroxide (0.03 mol), and in aqueous solution was treated with orthoboric acid to reduce the p.H. of the resin from about 8.2 to within the range 7.0 to 7.5. To this resin was added a conventional melamine formaldehyde resin (1 mol melamine to 2.75 mol formaldehyde), also catalysed with sodium hydroxide (0.0025 mol), and in aqueous solution in the proportion of one part melamine resin to two parts of phenol resin based on the solids content of the resins. 5% sodium metaborate, calculated on the total solids of the phenolic melamine resin mixture were then added. This composition in solution was used to impregnate sheets of 10 mil Kraft paper so that the paper contained between 33 and 38%, by weight of the resin impregnated paper, of the resinous composition solids. After a drying treatment to remove the solvent and cure the resin to a controlled extent, the paper was assembled as the core of a decorative laminated plastic material, and was surfaced with decorative printed sheets treated with urea melamine formaldehyde resins which in turn were surfaced by layers of alpha cellulose paper treated with melamine formaldehyde resin. After subjecting the assembly to heat and pressure to consolidate the sheets into a unitary product and to cure the resins, a laminated plastic sheet having Class 1 flame resistant properties according to British Standard Specification No. 476 was produced.

WHAT WE CLAIM IS:—

1. A process for producng a flame-resistant resinous composition which consists in adding to a water-containing solution of an alkali-catalysed resinous condensation product of formaldehyde and a phenol, a neutralising agent which gives rise, on reaction with free alkali, to inorganic salts having flame-resistant properties, and an amino formaldehyde resinous condensation product, the neutralising agent being added in an amount sufficient to neutralise the alkali-catalysed resinous condensate product of formaldehyde and a phenol.

2. A process as claimed in claim 1 in which the amount of flame resistant inorganic salt present after neutralisation is increased by the addition of one or more water soluble salts having flame resistant properties.

3. A process as claimed in claim 1 or 2 in which the proportion of an amino form-

aldehyde condensate to phenolic condensate based on solids is at least one to two.

4. A process as claimed in claim 1, 2 or 3 in which the alkali used to catalyse the phenolic condensation product is sodium hydroxide.

5. A process as claimed in claims 2-4 in which the neutralising agent is orthoboric acid.

6. A process as claimed in any of the preceding claims in which the amino compound used in the amino formaldehyde condensate is melamine.

7. A process as claimed in any of the preceding claims 2-6 in which the water soluble inorganic salt is added in a proportion exceeding 2.5% of the total solids of the resinous composition, the said salt preferably being sodium metaborate.

8. A process as claimed in any of the preceding claims in which the phenolic condensation product is substantially compatible with water.

9. Flame resistant laminated plastics comprising a core of fibrous material sheets, such e.g. as paper, fabric or asbestos or any combination thereof, treated with a resinous flame resistant composition produced by the process claimed in any of claims 1-8 before the core sheets are assembled and consolidated with the separate components of the laminated product.

10. Flame resistant laminated plastics as claimed in claim 9 in which the treated core sheets contain between 33 and 38%, by weight of the treated core sheets, of the flame resistant resinous composition.

11. A process for producing flame resistant resinous compositions substantially as hereinbefore described.

12. Flame resistant laminated plastics substantially as hereinbefore described.

For the Applicants,
CARPMAELS & RANSFORD,
Chartered Patent Agents,
24 Southampton Buildings, Chancery Lane,
London, W.C.2.

PROVISIONAL SPECIFICATION

Flame Resistant Materials

WE, FORMICA INTERNATIONAL LIMITED, a Company organised under the laws of Great Britain, of De La Rue House, 84-86 Regent Street, London, W.1., do hereby declare this invention to be described in the following statement:—

This invention relates to resinous compositions having flame resistant properties and to materials treated with such compositions.

Laminated plastics comprising fibrous materials treated with synthetic resins are widely usel in the construction of furniture of all kinds and as wall panelling. Although such materials are not inflammable and do not present a fire risk in themselves, it is desirable that they should have a high flame resistance particularly when they are used as constructional elements in buildings and ships.

The object of this invention is to provide resinous compositions which, when used to treat normally combustible fibrous materials, give rise to products having excellent flame resistant properties and more particularly, but not exclusively to those having Class 1 flame resistance characteristics as defined in British Standard Specification No. 476.

According to this invention a process for producing a flame resistant resinous composition comprises neutralising an alkali-catalysed resinous condensation product of formaldehyde and phenol or other phenolic compound capable of taking part in a condensation reaction with formaldehyde, and adding thereto an amino formaldehyde resinous condensation product and a water soluble inorganic salt having flame resistant properties.

Preferably the proportion based on solids of amino formaldehyde condensate to phenolic condensate is at least one to two.

Preferably the alkali used to catalyse the phenolic condensation product is sodium hydroxide.

Preferably the acid used to neutralise the phenolic condensation product is one giving rise to inorganic salts having flame resistant properties on reaction with free alkali, and is preferably orthoboric acid.

Preferably the amino compound used in the amino formaldehyde condensate is melamine.

The water soluble inorganic salt having flame resistant properties is preferably added in a proportion exceeding 2.5% of the total solids of the resinous composition, and is preferably sodium metaborate.

Preferably the phenolic condensation product is substantially compatible with water.

Flame resistant laminated plastics may be produced by treating at least the paper, fabric or other fibrous material core sheets for use in their manufacture with the resinous flame resistant composition before assembly and consolidation of the separate components of the laminates.

Preferably the core sheets contain between 33% and 38% by weight of the flame resistant resinous composition.

Although the phenolic resins may be catalysed by ammonium hydroxide, it is preferred to use those catalysed by sodium hydroxide because they are generally more compatible with water. In the treatment of

fibrous materials generally, and cellulosic materials in particular, substantially complete penetration with the resinous composition is required if they are to have uniform flame resistant properties and it is for this reason that water soluble resins are best. However, the solvent can include small quantities of methylated spirits.

The existence of free alkali-catalysed resins used in the production of laminated plastics detracts from the water resistance of the final product and for this reason it is necessary for it to be neutralised. Any acid can be used, but it is preferred to use an acid which will react with the free alkali and produce an inorganic salt having flame resistant properties and hence Orthoboric acid may be used but has the disadvantage of causing resin precipitation and resolubli-sation is necessary by the addition of methylated spirits or another suitable solvent.

It has been found necessary to add sodium metaborate to the resinous composition in order to increase the flame resistant characteristics which are not adequately provided by the borates produced by neutralisation of the sodium hydroxide. Other suitable salts, for example sodium pentaborate, may be used to replace the sodium metaborate. Flame resistant properties of materials treated with the resinous compositions according to this invention are good, but if Class 1 flame resistant characteristics, as defined in British Standard Specification No. 476, are required it is necessary to add not less than 2.5% and preferably about 5%, of the total resin solids, of the salt having flame resistant properties. We have found that about 5% of the salt is an optimum quantity.

In order to obtain the best flame resistant properties it is necessary to include in the composition amino formaldehyde condensation product resins because these are inherently more flame resistant than phenolic resins. Melamine formaldehyde resins are eminently satisfactory and although, for laminating compositions, the best proportion of melamine resin to phenolic resin is one part of the former to two parts of the latter, satisfactory products can be produced by using for example one part of phenolic resin or even two parts of melamine resin to one part of phenolic resin.

Again although phenol formaldehyde resin is eminently satisfactory, condensation products of cresols and formaldehyde can be used, provided the degree of condensation is controlled to produce products having high water compatibility.

In a particular case a conventional phenol formaldehyde resin catalysed with sodium hydroxide was treated with orthoboric acid to reduce the p.H. of the resin from about 8.2 to within the range 7.0 to 7.5. To this condensation product resin was added a conventional melamine formaldehyde resin also catalysed with sodium hydroxide in the proportion of one part melamine resin to two parts of phenol resin based on the solids content of the resins. 5% sodium metaborate calculated on the total solids of the phenolic melamine resin mexture were then added. This composition in solution was used to impregnate sheets of 10 mil Kraft paper so that the paper contained between 33 and 38% by weight of the resinous composition solids. After a drying treatment to remove the solvent and cure the resin to a controlled extent, the paper was assembled as the core of a decorative laminated plastic material, and was surfaced with decorative printed sheets treated with urea melamine formaldehyde resins which in turn were surfaced by layers of alpha cellulose paper treated with melamine formaldehyde resin. After subjecting the assembly to heat and pressure to consolidate the sheets into a unitary product and to cure the resins a laminated plastic sheet having Class 1 flame resistant properties according to British Standard Specification No. 476 was produced.

CARPMAELS & RANSFORD,
Agents for Applicants,
24 Southampton Buildings, Chancery Lane,
London, W.C.2.

Berwick-upon-Tweed: Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd.—1962
Published at The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.